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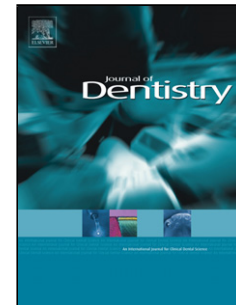
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Physico-mechanical characteristics of commercially available bulk-fill composites

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ABSTRACT

OBJECTIVES: Bulk-fill composites have emerged, arguably, as a new “class” of resin-based composites, which are claimed to enable restoration in thick layers, up to 4mm. The objective of this work was to compare, under optimal curing conditions, the physico-mechanical properties of most currently available bulk-fill composites to those of two conventional composite materials chosen as references, one highly filled and one flowable “nano-hybrid” composite.

METHODS: Tetric EvoCeram Bulk Fill (Ivoclar-Vivadent), Venus Bulk Fill (Heraeus-Kulzer), SDR (Dentsply), X-tra Fil (VOCO), X-tra Base (VOCO), Sonic Fill (Kerr), Filtek Bulk Fill (3M-Espe), Xenius (GC) were compared to the two reference materials. The materials were light-cured for 40 s in a 2x2x25 mm Teflon mould. Degree of conversion was measured by Raman spectroscopy, Elastic modulus and flexural strength were evaluated by three point bending, surface hardness using Vickers microindentation before and after 24 h ethanol storage, and filler weight content by thermogravimetric analysis. The ratio of surface hardness before and after ethanol storage was considered as an evaluation of polymer softening. Data were analyzed by one-way ANOVA and post-hoc Tukey’s test ($p=0.05$).

RESULTS: The mechanical properties of the bulk-fill composites were mostly lower compared with the conventional high viscosity material, and, at best, comparable to the conventional flowable composite. Linear correlations of the mechanical properties investigated were poor with degree of conversion ($0.09 < R < 0.41$) and good with filler content ($R > 0.8$). Softening in ethanol revealed differences in polymer network density between material types.

SIGNIFICANCE: Given the lower mechanical properties of most bulk-fill materials compared to a highly filled nano-hybrid composite, their use for restorations under high occlusal load is subject to caution. Further, the swelling behavior of some of the bulk-fill materials may be a reason for concern, which highlights the critical requirement for a veneering material, not only to improve aesthetic quality of the translucent material, but to reduce the impact of degradation.

INTRODUCTION

Due to considerable improvement since their inception, the use of photopolymerizable resin-based composite restorative materials are more frequently extended to large and deep cavities albeit with variable success (1, 2). In such cases, incremental build-up of multiple thin layers are required; first because of the limited cure depth (3, 4) and second to potentially reduce the consequences of shrinkage stress (5), although the latter theory has been refuted (6). However, layering techniques and multiple curing regimens of resin composites are time consuming. As a consequence, the composite material market is often driven by consumer demand for faster and easier procedures (sometimes at the cost of fundamental materials science principles) by reducing the curing time and/or using thicker composite layers. A modern example has seen the increasing popularity amongst dental practitioners of so-called “bulk-fill” materials, which are claimed to enable the restoration build-up in thick layers, up to 4mm. This new material class includes flowable and higher viscosity paste material types.

There currently exists a growing trend in the use of bulk-fill materials amongst practitioners due to a more simplified procedure. However, the lack of available literature on their clinical performance promotes much *in vitro* research, which ranks the properties of bulk-fill materials relative to the conventional flowable and paste composite types already on the market. In the available literature, some interesting characteristics were reported for bulk-fill materials. First, the possibility of adequately light-curing these materials to greater than 4-mm thickness was confirmed by microhardness measurements for X-traFil (VOCO) (7), SDR (Dentsply) (8, 9), Venus Bulk Fill (Heraeus-Kulzer) (8), Tetric EvoCeram Bulk-Fill (Ivoclar-Vivadent) and X-tra Base (VOCO) (10), and by measurements of degree of conversion for X-tra Base and SDR (9). However, the use of such methods to assess the quality of cure in depth may lead to an overestimation of the depth of cure (11). Specifically regarding bulk-fill composites, various depths of cure have been reported depending on the method used. The median depth of cure after 20 s cure of SDR, Venus Bulk-fill and Tetric EvoCeram Bulk-Fill was respectively 4.93, 6.08 and 3.83 mm when based on the ISO4049 method, and 2.5, 4.0 and 0.2 mm based on microhardness (12). Similarly, the extent of cure through depth indirectly evaluated by biaxial flexure strength measurements was significantly lower (< 4 mm) than when relying on degree of conversion or microhardness (9).

SDR was also associated with low shrinkage stress and shrinkage stress rates compared with various conventional pastes and flowable composites (9, 13). Similar observations were made for Tetric EvoCeram Bulkfil, Venus Bulk Fill, X-tra Fil, and Filtek Bulk-Fill (3M-ESPE) compared with Filtek Z250 (3M-ESPE) (14). In other work, SDR levels of shrinkage stress were lower than Filtek Supreme XT and Clearfil Majesty Posterior (Kuraray), but in the same range as Venus Diamond (Heraeus-Kulzer), Filtek Silorane (3M-ESPE), an experimental ormocer from VOCO or ELS (Saremco) (15). Nevertheless, despite the reduction in shrinkage stress, the advantages in terms of marginal adaptation are unclear. X-tra Fil (VOCO) was shown to present no significant difference in the amount of cuspal deflection compared to Filtek Supreme Plus (3M-ESPE) when filling a 4 mm-deep cavity in one step (7). Bulk-filling a 3.5 mm-deep cavity with X-tra Base and SDR was associated with significantly reduced cuspal deflection compared with a conventional composite, GrandioSO (VOCO) used in an oblique incremental filling technique, although no associated change in cervical microleakage was observed (16). Similarly, equivalent marginal adaptation was observed when SDR was used as a base under conventional hybrid composites compared to the latter used incrementally (17). SDR was shown to significantly improve microtensile bond strength compared to Filtek Z100 (3M-ESPE) and G-aenial Universal Flo (GC) when filling cavities of high C-factor in bulk, but no difference in bond strength was observed when an incremental filling technique was employed or when bonding to a low C-factor surface (18).

The main advancements of bulk-fill materials, namely increased depth of cure, which probably results from higher translucency (19), and low shrinkage stress are related to modifications in the filler content and/or the organic matrix. Ideally, these perceived improvements should not be at detriment to the mechanical properties of the material. Recent studies have reported that bulk-fill resin composites exhibited acceptable levels of creep resistance, in the range shown by conventional material types (20, 21), although some bulk materials investigated (SDR and Venus Bulk Fill) presented a significantly higher creep strain than the nanohybrid composite Filtek Supreme XT (3M-ESPE) (21). Among flowable composites (EsthetX Flow, Dentsply; Filtek Supreme Plus Flow, 3M-ESPE), SDR exhibited the lowest Vickers hardness, the highest elastic modulus and the highest creep, but all three properties were much lower than hybrid

composites (Filtek Silorane, 3M-ESPE; EsthetX Plus, Dentsply; Filtek Supreme Plus, 3M-ESPE) (13). Similarly, another study raised some concerns regarding low to very low hardness and elastic modulus for some bulk-fill materials, especially SDR, Venus Bulk Fill and Filtek Bulk-Fill (22). In other work, some improvement in elastic modulus, flexural strength and greater increase in fracture toughness were attributed to a bulk-fill material containing glass microfibers (Xenius, GC) compared with bulk-fill types (23).

The objective of the present work was to group all the main currently available bulk-fill composites as well as a dual-cure composite in a single study (Table 1), and to compare their physico-mechanical properties under optimal curing conditions to those of two conventional composite materials chosen as references, one highly filled and one flowable nano-hybrid composite: Grandio and Grandio Flow (VOCO). The null hypothesis was that there are no differences in physico-mechanical properties between neither of the so-called bulk-fill composites, nor with two conventional composite materials chosen as controls.

MATERIALS & METHODS

The materials used in the present investigation are presented in Table 1. They were placed in a 2x2x25mm Teflon mould and light-cured by four 40 s overlapping irradiations on the upper sample side to ensure optimal mechanical properties. The light tip of the polywave LED light BluePhase G2 (Ivoclar-Vivadent, Schaan, Liechtenstein) was placed against a polyester film at the upper sample surface in order to minimize the effects of oxygen inhibition and polymerization was initiated using the high-power irradiation mode (1050 mW/cm^2 , measured by Bluephase Meter, Ivoclar-Vivadent, Schaan, Liechtenstein). After photopolymerization, the samples were carefully removed from the mold and stored dry for 24 h in the dark at room temperature ($23 \pm 1^\circ\text{C}$) before analysis, to ensure that the polymerization process was complete prior to analysis (24, 25).

The elastic modulus (E_{mod}) and flexural strength (σ_f) were measured using a three-point bend test. Samples ($n=5$) were loaded in a universal testing machine (Instron 5566, High Wycombe, UK) at a strain rate of 0.75 mm/min until fracture occurred as recommended in ISO4049 and as previously described (26).

Vickers microhardness measurements were carried out on the fractured samples recovered from the previous analyses (Dry VHN) ($n=5$). A 200 g load was applied for 30 s on the upper surface using a Durimet microhardness tester (Leitz, Wetzlar, Germany). Since that surface was in direct contact with a polyester film providing a uniform surface lustre, no polishing was performed. The length of the diagonal of each indentation was measured directly using a graduated eye-lens. The Vickers Hardness Number was calculated as previously described (26). The same samples were then immersed in pure ethanol for 24 h, before re-measuring the microhardness (Ethanol VHN). The ratio between ethanol VHN and dry VHN (%) was used as an indication of network density.

Thermogravimetric analysis (TGA/SDTA861e, Mettler-Toledo, Greifensee, Switzerland) was used to determine the filler mass fraction. The resin composites were subject to a temperature rise from 30 to 900°C at the rate of 10°C/min followed by air-cooling to room temperature. The inorganic fraction was determined by the ratio of the final and initial sample mass ($n=3$).

The degree of conversion (DC, in %) was measured on the upper sample surface (n=3) using a Raman Spectrometer (DXR Raman Microscope, Thermo Scientific, Madison, WI USA). The samples were excited at 780 nm by a frequency-stabilized single mode diode laser through a microscope objective (x 50) and spectra were obtained in the region 1600 cm^{-1} , with the following conditions: microhole, 50; irradiation time, 60 s; number of accumulations, 5; grating 400 lines/mm. The DC was then calculated based on the decrease in intensity of the peak corresponding to the methacrylate C=C groups at 1640 cm^{-1} compared to the uncured sample; the aromatic peak at 1610 cm^{-1} was used as the internal standard.

Data were analyzed by one-way ANOVA and Tukey's test ($p=0.05$). Multivariate linear regression analysis was performed to study the relationship between the investigated properties.

RESULTS

Filler mass fractions range from 60.7 to 85.3 for bulk-fill composites (Figure 1). Only two materials, X-traF and SonicF, contain significantly more fillers than GrandioFlow, and only X-traF has filler levels comparable to Grandio. DC are presented in Figure 2, and range between 43.6 and 76.5 %. DC are in the range of the controls for a majority of materials, except for FiltekBF, significantly lower (43.6 %, $p>0.05$) and SDR (67.6 %; $p>0.05$ versus GrandioF only), VenusBF and SonicFill (71.2 and 76.5 %, respectively; $p>0.05$ versus Grandio and GrandioF), significantly higher. Regarding E_{mod} , two approximate groups of bulk-fill materials can be highlighted based on the statistics, first those with comparable or higher E_{mod} than GrandioFlow, and second those with significantly lower E_{mod} (Figure 3). Grandio presents a significantly higher E_{mod} than all materials. As regards σ_f , the values range from 76.0 to 140.3 MPa, and the statistical associations are less discriminative than for E_{mod} (Figure 4). Only three materials, i.e. FiltekBF, ColDCBF, and VenusBF, have significantly lower σ_f than both controls. Finally, microhardness values seem to be the most discriminative between the investigated materials, dry VHN ranging from 21.7 to 120.8 (Figure 5a), and ethanol VHN from 6.0 to 99.0 (Figure 5b). Grandio displays significantly higher VHN (dry and ethanol) than all other materials, while a group of three materials display very low dry and ethanol VHN: FiltekBF, SDR and VenusBF. Only SonicF and X-traF compete with the values of GrandioFlow, the values of the other materials being significantly lower. As for the ratio between dry and ethanol VHN (Figure 5c), half of the materials including both controls display high ratios (82.2 to 90 %), while the ratios of the other half ranges from 68.7 % down to 19.2 % for SDR. The linear correlation coefficients of multivariate correlations between the investigated variables are reported in Table 2. The latter indicates several good linear correlations, notably between mechanical properties and filler fraction ($R > 0.8$). On the contrary, DC was poorly correlated with the mechanical properties ($0.09 < R < 0.41$).

DISCUSSION

Large and significant differences ($p < 0.001$) were observed for all considered physico-mechanical properties (Filler mass fraction, DC, E_{mod} , σ_f , dry VHN, ethanol VHN and their ratio) within the bulk-fill composite category as well as with the two conventional composites chosen as controls, which led to the rejection of the null hypothesis.

As mentioned by El-Safty *et al.* (21), bulk-fill composite materials are likely to fulfill some important requirements, notably low polymerization shrinkage, ease of use, improved depth of cure (≥ 4 mm) and enhanced physical characteristics. The latter is particularly important since bulk-fill composites will represent most, if not all of the restoration. According to the present work, the mechanical properties of the bulk-fill composites are mostly lower than Grandio and at best comparable to those of GrandioFlow (Figures 2-5) and in this regard, they seem to exhibit properties closer to flowable materials than to micro- or nano-hybrid composites (27). Since flowable materials are never recommended to represent most of the restoration bulk, it is questionable whether this should be the case for bulk-fill composites, and their use for restorations under high occlusal load should remain subject to caution.

Grandio has been ranked in several studies among the best commercial materials in terms of hardness, flexural strength and elastic modulus (26-30), predominantly due to its high filler content. Similarly, GrandioFlow with the highest mechanical properties of competitor flowable composites is also stronger in many instances than hybrid paste composites (27, 29, 31). The small reduction of the properties of Grandio after ethanol storage was documented by (28), and is confirmed by the present microhardness results (Figure 5). Hence, the rationale for choosing these materials as control was that they both present the highest mechanical performances in their respective categories. Besides, they were previously used in other works by our group and therefore serve as an internal standard. Although, in the present investigation, the bulk-fill materials exhibited lower mechanical properties compared with the highly filled (control) composites, it should be noted that the properties of some bulk-fill composites may be equivalent to other, more conventional composites on the market. For example, Tetric EvoCeram Bulk-Fill presents close properties to those of its conventional counterpart from the same manufacturer, Tetric EvoCeram ($E_{mod} \sim 6-7$ GPa, $\sigma_f \sim 90$ MPa, VHN ~ 50) (26). However, in numerous other instances, bulk-fill materials may match the performance of a conventional

composite for a certain property, and not for others. For example, Filtek BF presents σ_f in the same range as Filtek Supreme XT (~90-100 MPa), but lower E_{mod} (~4 compared with 7-8 GPa, respectively) and microhardness (~30 compared with 60-80 VHN, respectively) (26, 29). Similarly, X-traFil and Grandio in the present work exhibit similar σ_f (~130 MPa) but significantly different E_{mod} (9 compared with 15 GPa, respectively) and microhardness (70 compared with 120 VHN, respectively). Besides, and as suggested here and in previous work, the veneering of a bulk-fill composite restoration using a conventional composite material is essential (22), and from the present results it is clear that this should not only be limited for aesthetic reasons.

The variability in mechanical properties within currently available materials that claim to be “bulk-fill” observed in the present study is also supported by previous data (22, 23). For example, X-traF and SonicF are frequently ranked highest in terms of strength characteristics and X-traB and Xenius exhibit reasonable mechanical properties, whereas SDR, VenusBF and Filtek BF often present the lowest values. According to the results of this study, variability in the results can be explained by the differences in filler content. Good linear correlations of mechanical properties and filler mass fraction were observed ($R > 0.8$, Table 2). Significant correlations ($R > 0.8$) between surface microhardness and filler mass fraction have also been previously reported (13, 26, 32). The positive correlation between E_{mod} and filler mass fraction is also in accordance with previous work (13, 33), but not in others. For example, in a study including different material technology (Ormocers), the influence of the organic matrix was more prominent, since the correlation between E_{mod} and filler mass fraction was significantly increased when the Ormocer composite was excluded (from $R=0.37$ to 0.70 ; (26)). Hence, some differences in mechanical properties may also be due to specificities of the organic matrix, such as variations of polymer network density. Indeed, for some materials, in particular VenusBF, SDR and Filtek BF, significant softening in ethanol reveals differences in polymer network density (Figure 5c). The possible use of plastifying monomers to reduce shrinkage stress may explain why these materials are prone to softening. Such an observation is obviously a cause for concern, and further supports their bulk volume being covered by another material. Other reasons for the differences in mechanical properties between the investigated materials, include increased particle size (e.g. SDR, X-traFil or X-traBase) compared with conventional resin composites (34). In addition, the use of other photoinitiators, such as

“Ivocerin” in Tetric EvoCeram Bulk-Fill, may affect the results as well. As mentioned in a previous paper, the comparison of commercial material properties is made difficult by the fact that other parameters such as filler size and morphologies, monomer type and ratio or photoinitiation chemistries vary greatly between products (26). Within the limitations of the present work, the only conclusion supported by our data is that filler mass fraction seems to be an important parameter governing the mechanical properties of the investigated materials. This is supported for example by the case mentioned earlier of Tetric EvoCeram Bulk-Fill and its conventional counterpart from the same manufacturer Tetric EvoCeram, which have very close E_{mod} , σ_f and VHN around 50 VHN, and in fact similar filler mass fraction (around 70%) (26). On the contrary, Grandio and X-traFil (also bulk and conventional counterparts, from another manufacturer) present similar σ_f but significantly different E_{mod} and microhardness, despite a similar filler mass fraction (around 85%), which may then relate to the involvement of the other parameters mentioned above (particle size and density, monomer type and ratio or photoinitiators).

Xenius (which is the previous version of the current Ever-X posterior) is the sole material of this work containing glass microfibers. This composite was previously reported to exhibit high fracture toughness as well as good flexural strength values and low shrinkage strain (23). However, despite the fact that the work includes nano-hybrid composites such as FiltekZ250 or FiltekSupremeXT, the mechanical properties of these materials do not appear in all charts. Despite that, the properties of Xenius common in both works seem to follow similar trends.

While usually considered as an important material parameter for a given material formulation, degree of conversion was poorly correlated with the mechanical properties in the present work ($0.09 < R < 0.41$). The low correlation between DC and mechanical properties should be expected, first because all materials are based on different monomer contents, and therefore present their own specific relationship between DC and mechanical properties. Second, all materials in this study were cured more than the manufacturers' recommendations (40 s), and it is therefore very likely that each material is optimally cured. Only with suboptimal cure can differences in DC result in differences in mechanical properties (11), again for a given material composition. The lack of correlation between DC and E_{mod} for bulk-fill composites is in

accordance with previous work including VenusBF and SDR, the former presenting at all depth significantly higher DC but significantly lower macro- and micromechanical properties (8). E_{mod} was clearly identified as an important factor affecting shrinkage stress of resin-based composites (35). The lower shrinkage stress, and in some cases improved marginal adaptation reported for bulk-fill materials, are probably to relate to their low to very low E_{mod} . It is interesting to note that among several bulk-fill composites, only X-traFil was not associated with a reduction in shrinkage stress by (14), which happens to be the most highly filled bulk-fill material in the present study, and the one with the highest E_{mod} . Hence, a compromise seems unavoidable: either reducing stress or maintaining high elastic modulus to withstand occlusal forces. According to a critical literature review on the mechanical properties of human dentin, E_{mod} values measured for dentin using mechanical testing should range between 12 and 20 GPa (36). Despite the difficulty to directly compare values obtained with different methods, it appears that, in the present work, only the hybrid composite is approaching the dentin values (15.5 GPa for Grandio), whereas the materials from the bulk-fill category present lower modulus values (3.3 to 9.4 GPa). While some bulk-fill materials like X-traF, SonicF or Xenius compete with the E_{mod} values reported for hybrid composites in the literature (~8 GPa) (27, 29, 37), others such as SDR, FiltekBF and VenusBF present lower values corresponding to those reported for flowable composites (~4 GPa) (27, 31, 37). As mentioned in the introduction, little or no interfacial improvement was clearly observed when using bulk-fill composite restoration compared to layered restoration with hybrid composite. It is therefore questionable whether we should switch from hybrid composite materials, which at best reach the lowest values of modulus reported for dentin, to materials with an even lower modulus to restore the majority or entirety of the lost tissue.

Finally, it is important to mention that the present values are measured under ideal laboratory conditions, i.e. higher irradiance, longer curing time and direct contact of the light-curing tip with the sample, which may not necessarily be expected in general practice. Both intrinsic and extrinsic factors are known to affect polymerization efficiency (39). In the case of bulk-fill materials, the impact of each specific compound on the final material properties is difficult to predict, since specific material composition is largely unknown. For example, the presence of alternative photoinitiators requires the use of broadband spectrum lights. For that reason, to

compare the materials on a fair basis, a polywave LED light was used in the present work, at high irradiance and long irradiation time (40 s). However, in less ideal conditions such as lower irradiance (due to an increased light tip to material distance, tip contamination and/or reduced power), a further reduction of material properties might be expected, again, dependent upon on the material considered as shown for Tetric Evo Ceram bulk and X-tra Base (10).

CONCLUSION

The reduction of time and improvement of convenience associated with bulk-fill materials is a clear advantage of this particular material class. However, a compromise with mechanical properties compared with more conventional commercially-available nano-hybrid materials was demonstrated by the present work. Given the lower mechanical properties of most bulk-fill materials compared to a highly filled nano-hybrid composite, their use for successful restorations under high occlusal load may be controversial. Besides, the significant decrease in surface hardness after ethanol storage of some of the bulk-fill materials investigated raises concern regarding long-term stability and suggests that these materials should be better prevented from direct contact with the oral cavity, which then, of course, reduces their convenience.

FIGURE CAPTIONS

Figure 1: Filler mass fraction (%) measured by TGA (n=3). The materials are ranked in descending order according to their means (black horizontal bars) and the standard deviations are added in the form of grey bars. Vertical bars connect materials that are not statistically different ($p>0.05$) (n=3).

Figure 2: Degree of conversion (%) measured by Raman spectroscopy. The materials are ranked in descending order according to their means (black horizontal bars) and the standard deviations are added in the form of grey bars. Vertical bars connect materials that are not statistically different ($p>0.05$) (n=5).

Figure 3: Elastic modulus (GPa) measured by three points bending. The materials are ranked in descending order according to their means (black horizontal bars) and the standard deviations are added in the form of grey bars. Vertical bars connect materials that are not statistically different ($p>0.05$) (n=5).

Figure 4: Flexural strength (MPa) measured by three points bending. The materials are ranked in descending order according to their means (black horizontal bars) and the standard deviations are added in the form of grey bars. Vertical bars connect materials that are not statistically different ($p>0.05$) (n=5).

Figure 5: Vickers microhardness (VHN) (a) after 24 hours of dry storage in the dark, (b) after 24 hours of storage in ethanol and (c) the ratio between both. The materials are ranked in descending order according to their means (black horizontal bars) and the standard deviations are added in the form of grey bars. Vertical bars connect materials that are not statistically different ($p>0.05$) (n=5).

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Table 1 – Characteristics of tested materials

Materials	Abbreviation	Manufacturer	Composite Type	Shade	Batch
Tetric Evo Ceram Bulk Fill	TECBF	Ivoclar-Vivadent (Schaan, Liechtenstein)	Bulk-fill paste composite	IVA	P63316
Venus Bulk Fill	VenusBF	Heraeus-Kuzer, (Hanau, Germany)	Bulk-fill flowable	U	10100
Surefil SDR Flow	SDR	Dentsply, (Konstanz, Germany)	Bulk-fill flowable composite	A3	120 3000 624
X-tra fil	X-traF	Voco (Cuxhaven, Germany)	Bulk-fill paste composite	U	1209605
X-tra base	X-traB	Voco (Cuxhaven, Germany)	Bulk-fill flowable composite	U	1208392
Sonic Fill	SonicF	Kerr (Orange, CA, USA)	Bulk-fill paste composite with sonic hand-piece	A3	3851500
Filtek Bulk Fill	FiltekBF	3M-Espe (St. Paul, MN, USA)	Bulk-fill flowable composite	U	N370958
Xenius (previous version of Ever-X posterior)	Xenius	GC Europe (Leuven, Belgium)	Bulk-fill paste composite with glass microfibres	B	20071108
Coltene Dual-cure Bulk-Fill	Col DCBF	Coltene-Whaledent (Altstätten, Switzerland)	Dual-cure Bulk-fill flowable composite	U	20071108
Grandio	Grandio	Voco (Cuxhaven, Germany)	Hybrid paste composite	A3	120983
Grandio Flow	GrandioF	Voco (Cuxhaven, Germany)	Hybrid flowable composite	A3	1208317

Table 2 – Multivariate correlation coefficients (Restricted maximum likelihood)

	Filler mass fraction	Dry VHN	Ethanol VHN	E_{mod}	σ_f	DC
Filler mass fraction	1.00	0.86	0.84	0.84	0.83	0.43
Dry VHN	0.86	1.00	0.96	0.97	0.65	0.19
Ethanol VHN	0.84	0.96	1.00	0.92	0.65	0.09
E_{mod}	0.84	0.97	0.92	1.00	0.66	0.20
σ_f	0.83	0.65	0.65	0.66	1.00	0.41
DC	0.43	0.19	0.09	0.20	0.41	1.00

Figure 1

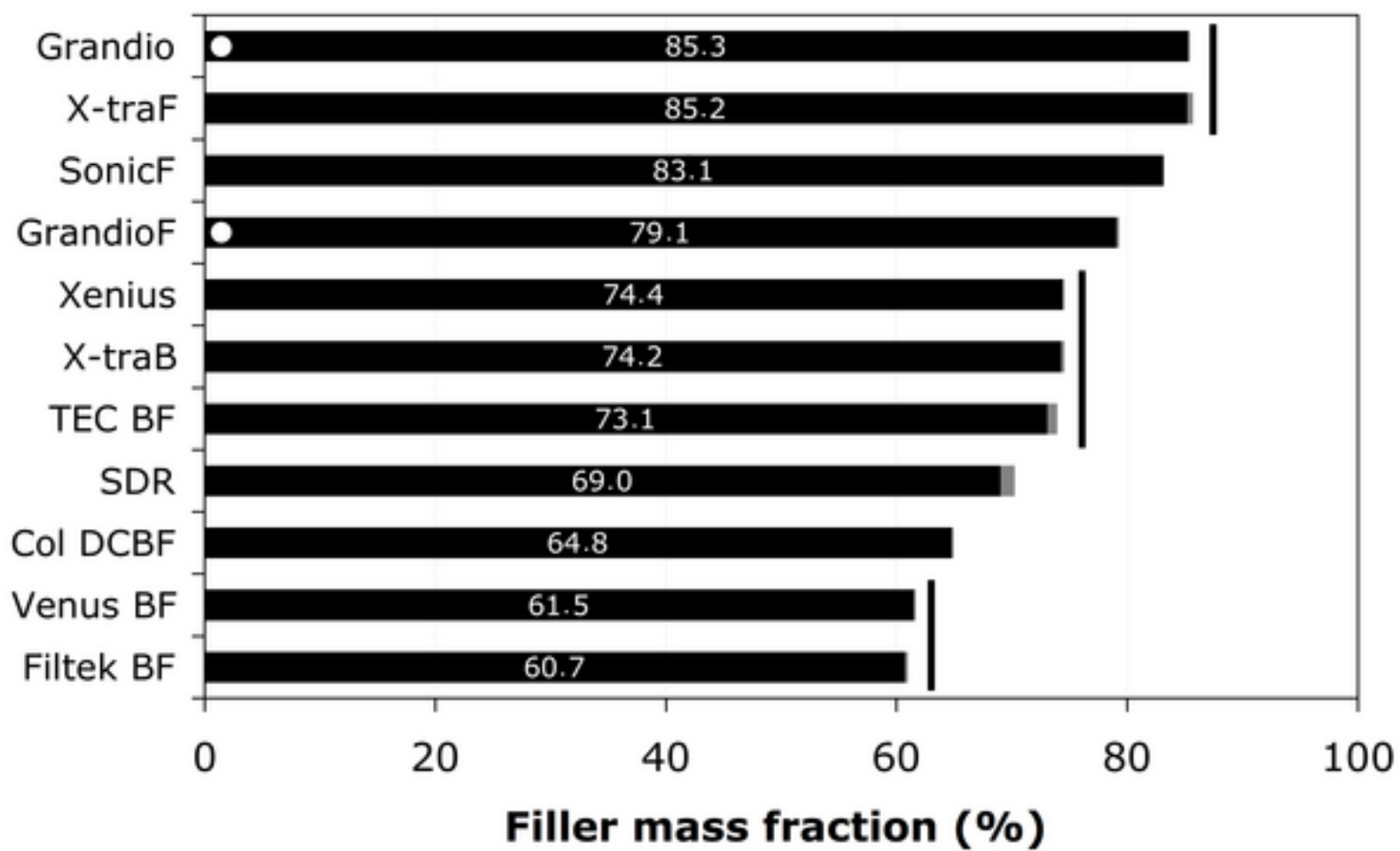


Figure 2

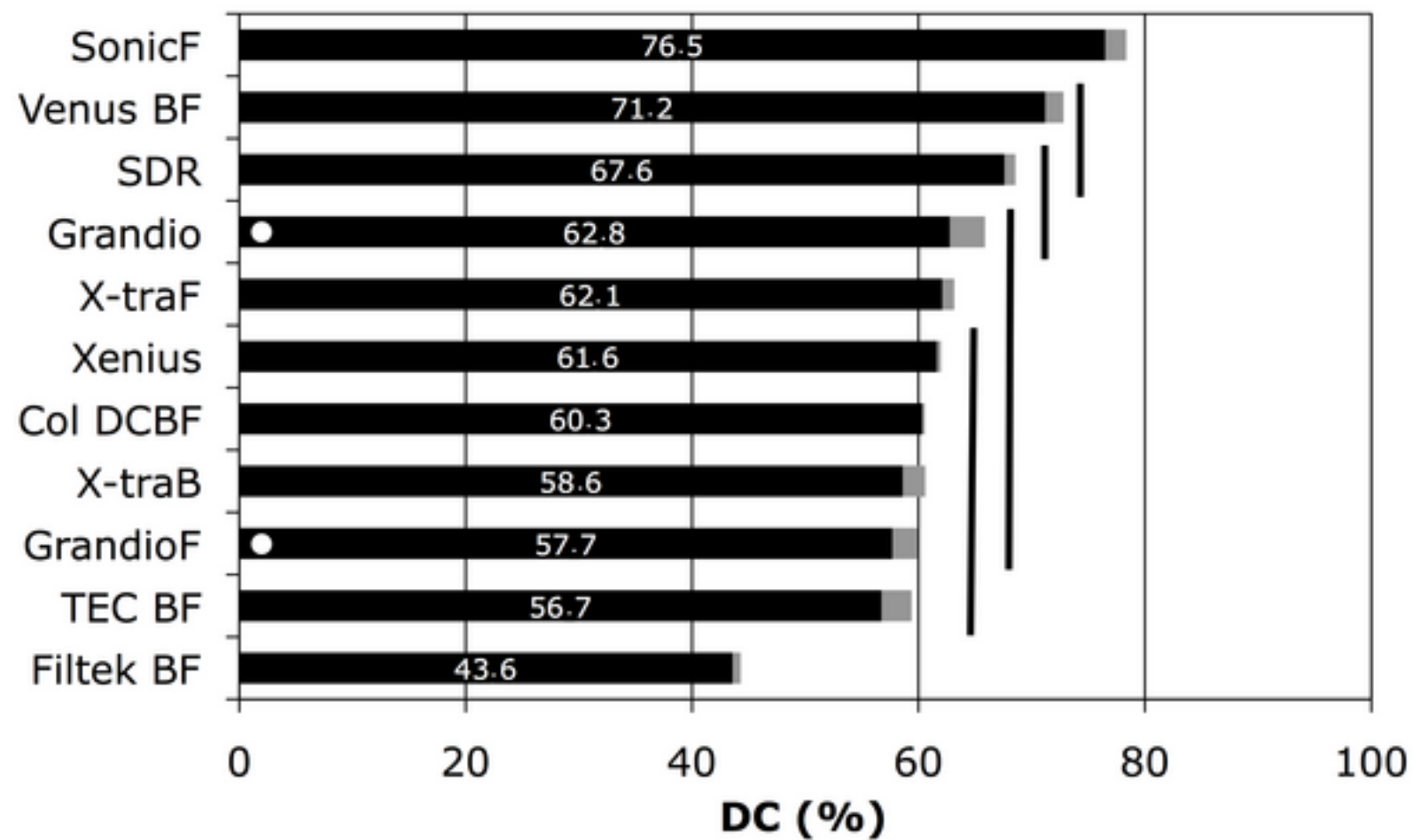


Figure 3

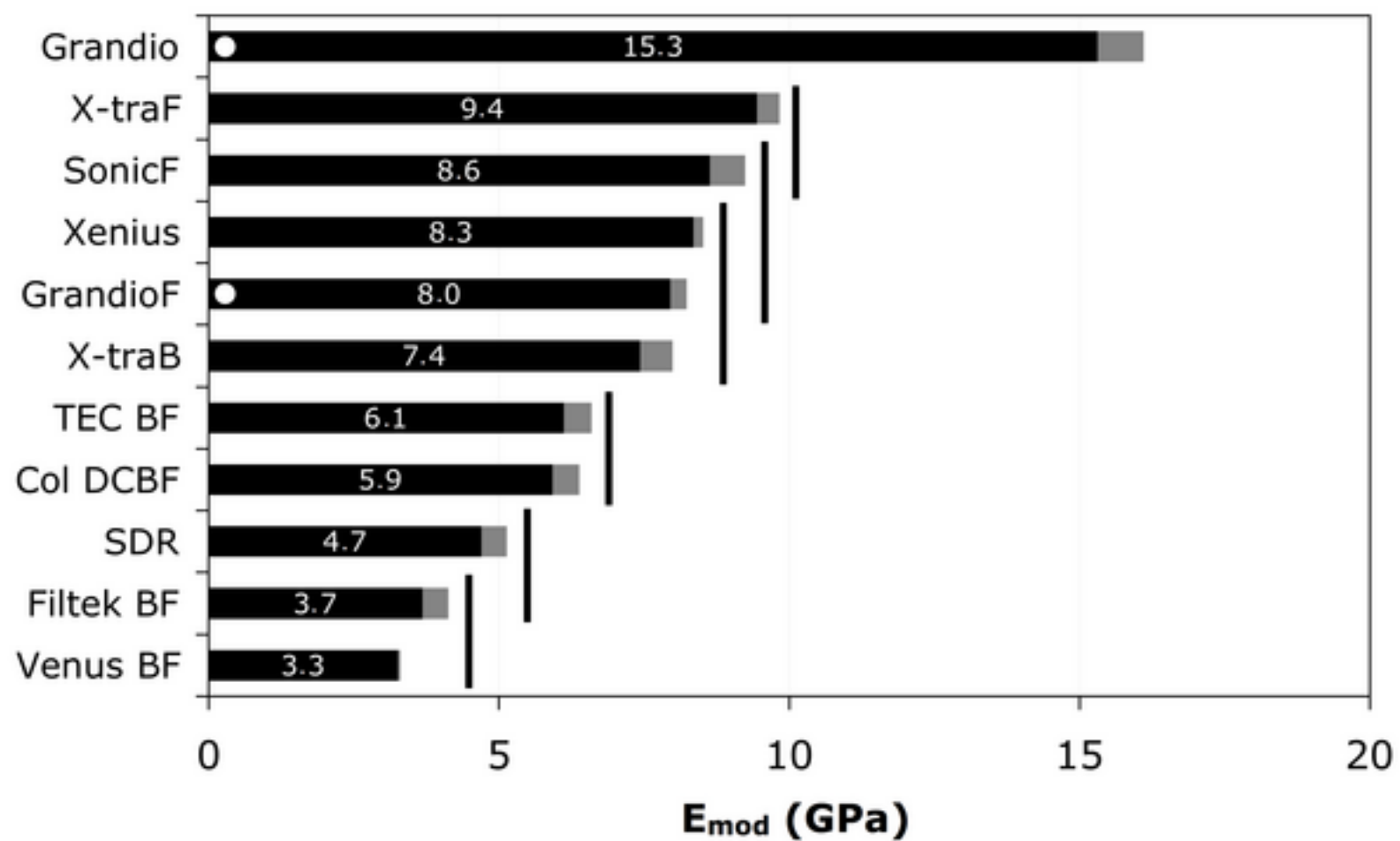
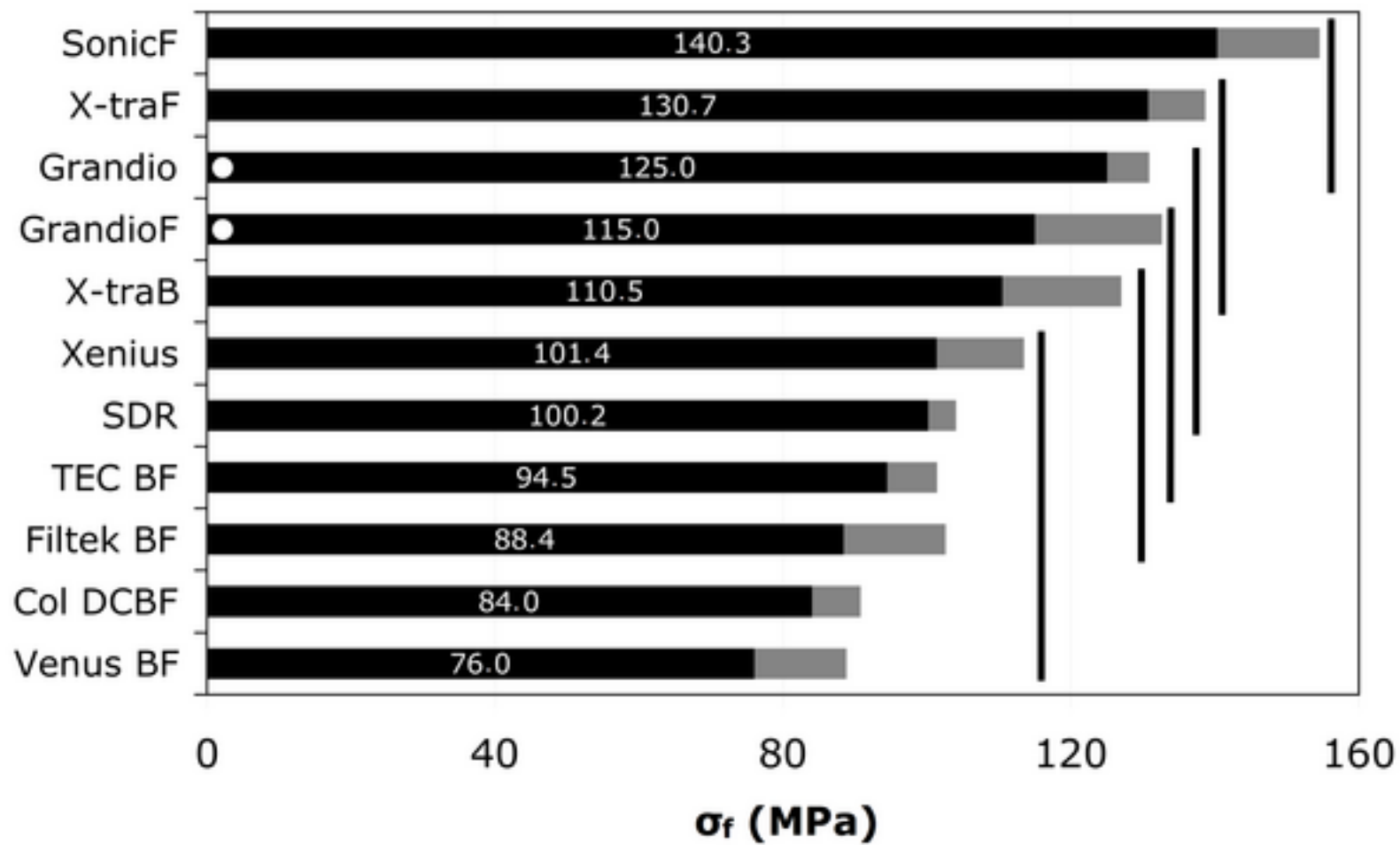
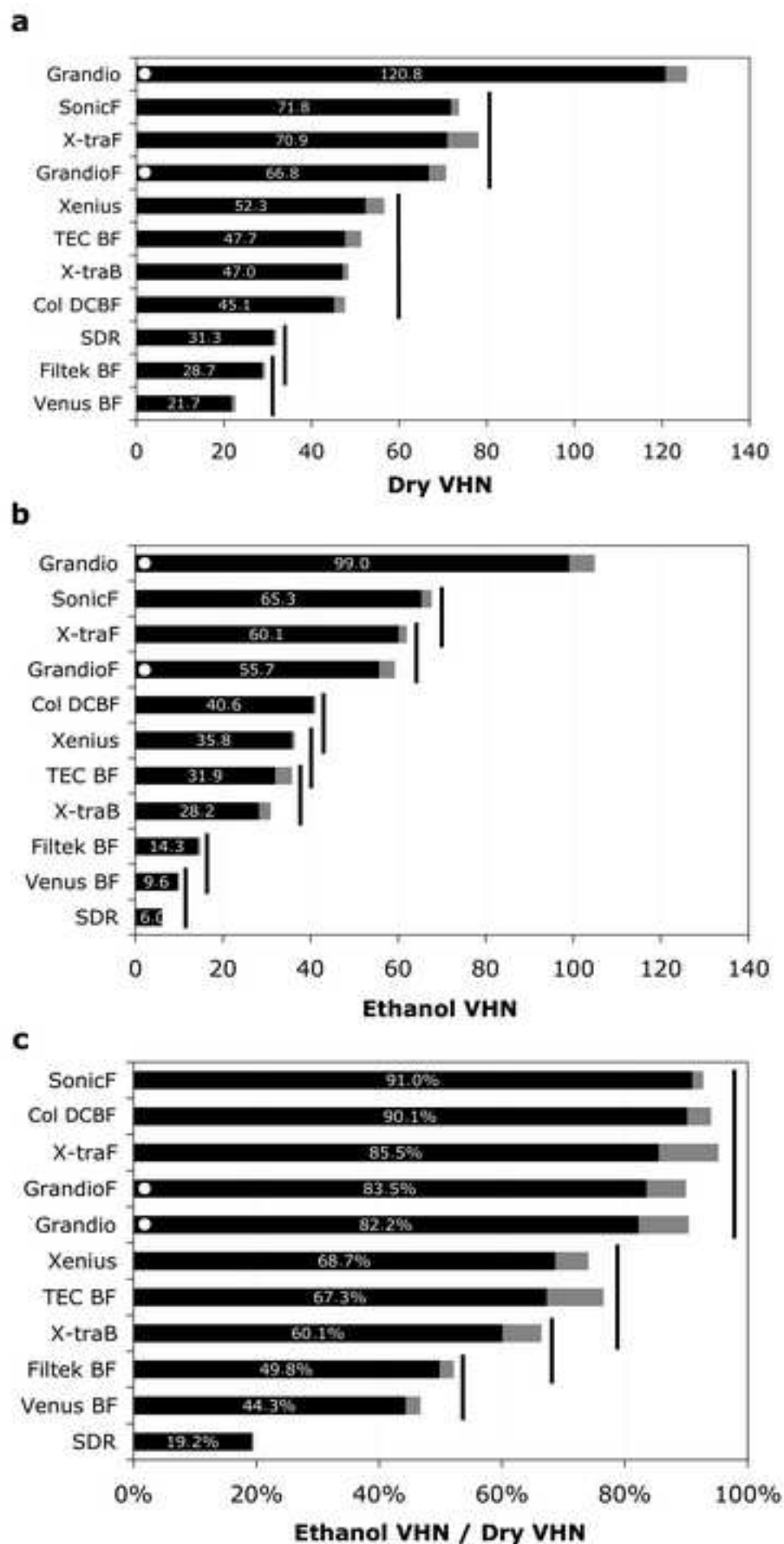


Figure 4





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